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(71) Applicant *(for all designated States except US)*: THE PROCTER & GAMBLE COMPANY [US/US]; One Procter & Gamble Plaza, Cincinnati, OH 45202 (US).

(72) Inventors; and
(75) Inventors/Applicants *(for US only)*: SURUTZIDIS, Athanasios [GR/US]; 6023 Bridgeton-Manor Drive, Hamilton, OH 45011 (US). LEBLANC, Michael, Jude [US/US]; 2216 Ravine Street, Cincinnati, OH 45219 (US).

(74) Agents: REED, T., David et al.; The Procter & Gamble Company, 5299 Spring Grove Avenue, Cincinnati, OH 45217 (US).

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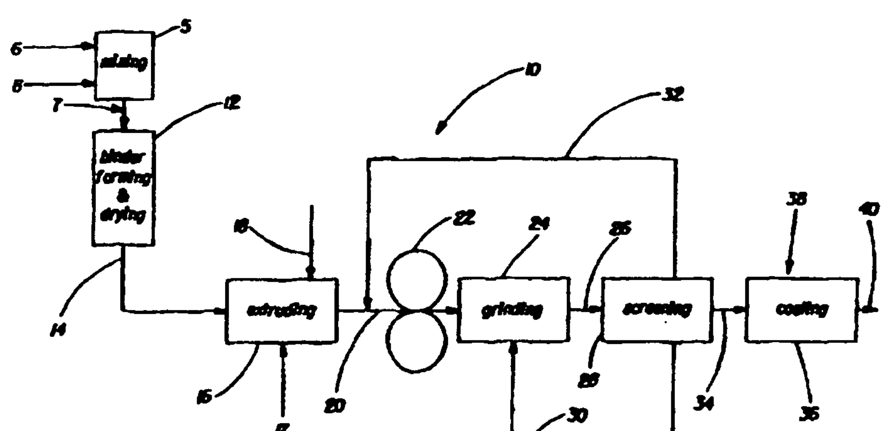

(57) Abstract

A process for producing a particulate laundry additive composition for perfume delivery primarily in laundry detergent and fabric softening products is disclosed. The aforementioned needs in the art are met by the present invention which provides a process for producing a particulate laundry additive composition for perfume delivery primarily in laundry detergent and fabric softening products. The process essentially includes the steps of inputting an aqueous mixture a first encapsulating material and porous carrier particles (e.g., zeolite X) into an extruder, and thereafter, extruding the first encapsulating material, which is preferably a glassy carbohydrate material, with porous carrier particles, preferably loaded with a perfume, so as to form a hot extrudate. Subsequently, the steps of cooling and grinding the extrudate into granules is completed. In the final step, a second encapsulating material is coated onto the granules to enrobe further the perfume in the carrier particles.

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DUAL COATING PROCESS FOR PRODUCING A PARTICULATE LAUNDRY ADDITIVE COMPOSITION FOR PERFUME DELIVERY HAVING IMPROVED PHYSICAL PROPERTIES

FIELD OF THE INVENTION

The present invention generally relates to a process for producing a particulate laundry additive composition, and more particularly, to an extrusion process which produces a dual-coated particulate laundry additive for perfume delivery in laundry detergent compositions, especially those in the form of granules, agglomerates, laundry bars or pastilles. This process improves upon existing processes in that it provides a composition having unexpectedly better physical properties such as appearance ("whiteness"), moisture protection and perfume protection as evidenced by its substantially reduced odor that is emitted by the particles. The process of the invention may also be employed to produce particulate additive compositions which may be used in fabric softening and dishwashing as well as laundry detergent compositions.

BACKGROUND OF THE INVENTION

Most consumers have come to expect scented laundry products and to expect that fabrics which have been laundered also have a pleasing fragrance. Perfume additives make laundry compositions more aesthetically pleasing to the consumer, and in some cases the perfume imparts a pleasant fragrance to fabrics treated therewith. However, the amount of perfume carryover from an aqueous laundry bath onto fabrics is often marginal. The detergent manufacturing industry, therefore, has long searched for an effective perfume delivery system for use in laundry products which provides long-lasting, storage-stable fragrance to the product, as well as fragrance to the laundered fabrics.

Laundry and other fabric care compositions which contain perfume mixed with or sprayed onto the compositions are well known in the art and currently commercialized. Because perfumes are made of a combination of volatile compounds, perfume can be continuously emitted from simple solutions and dry mixes to which the perfume has been added. Various techniques have been developed to hinder or delay the release of perfume from compositions so that they will remain aesthetically pleasing for a longer length of time. To date, however, few of the methods deliver significant fabric odor benefits after prolonged storage of the product.

Moreover, there has been a continuing search for methods and compositions which will effectively and efficiently deliver perfume from laundering solutions onto fabric surfaces. As can be seen from the following disclosures in the prior art, various methods of perfume delivery have been dev loped involving protection of the perfume through

the wash cycle, with release of the perfume onto fabrics. For example, one method entails delivering fabric conditioning agents, including perfume, through the wash and dry cycle via a fatty quaternary ammonium salt. Another method involves a microencapsulation technique which involves the formulation of a shell material which will allow for diffusion of perfume out of the capsule only at certain temperatures. Yet another method involves incorporating perfume into waxy particles to protect the perfume through storage in dry compositions and through the laundry process. The perfume allegedly diffuses through the wax on the fabric in the dryer. Further prior art disclosures involve perfume dispersed with a water-insoluble nonpolymeric carrier material and encapsulated in a protective shell by coating with a water-insoluble friable coating material, and a perfume/cyclodextrin complex protected by clay which provides perfume benefits to at least partially wetted fabrics.

Still another method for delivery of perfume in the wash cycle involves combining the perfume with an emulsifier and water- soluble polymer, forming the mixture into particles, and adding them to a laundry composition. The perfume can also be absorbed onto a porous carrier material, such as a polymeric material. Perfumes have also been absorbed onto a clay or zeolite material which is then admixed into particulate detergent compositions. Generally, the preferred zeolites have been Type A or 4A Zeolites with a nominal pore size of approximately 4 Angstrom units. It is now believed that with Zeolite A or 4A, the perfume is absorbed onto the zeolite surface with relatively little of the perfume actually absorbing into the zeolite pores.

While the adsorption of perfume onto zeolite or polymeric carriers may perhaps provide some improvement over the addition of neat perfume admixed with detergent compositions, industry is still searching for improvements in the length of storage time of the laundry compositions without loss of perfume characteristics, in the intensity or amount of fragrance delivered to fabrics, and in the duration of the perfume scent on the treated fabric surfaces. Furthermore, even with the substantial work done by prior skilled artisans in this area, a need still exists for a simple, more efficient and effective perfume delivery system, preferably in particulate form, which can be mixed with laundry compositions to provide initial and lasting perfume benefits to fabrics which have been treated with the laundry product.

Another problem associated with perfume delivery systems, especially those in particulate form, is concerned with the method by which such particulate perfume delivery systems are made. It has been difficult to produce perfume delivery systems particularly those involving zeolite or polymeric carriers in an economic and efficient manner. Oftentimes, a significant am unt of the perfume will vaporate from the carrier material during processing as well as during storage prior to use. Additionally, many

materials which are included in the perfume delivery system to prevent the volatilization of perfume prior to deposition on fabrics can degrade during manufacture, thereby losing their effectiveness. As a consequence, the particulate perfume delivery system sometimes has a tendency to emit its perfume component while stored in the final product, thereby interfering with the desired odor of other perfumes contained in the product. Thus, there has been a need for not only an effective perfume delivery system or additive for laundry detergents, but for a process which can produce such a laundry perfume delivery additive which is efficient, economical, and minimizes or eliminates evaporation of perfume and degradation of materials used during processing.

Accordingly, despite the aforementioned disclosures in the art, there remains a need for a process for producing a particulate laundry additive composition for perfume delivery in laundry detergent and other cleaning or fabric softening products. Additionally, there is a need for such a process which is not only more economical and efficient, but also minimizes evaporation of perfume and the degradation of materials used in this regard during production.

BACKGROUND ART

U.S. Patent 4,539,135, Ramachandran et al, issued September 3, 1985, discloses particulate laundry compounds comprising a clay or zeolite material carrying perfume. U.S. Patent 4,713,193, Tai, issued December 15, 1987, discloses a free-flowing particulate detergent additive comprising a liquid or oily adjunct with a zeolite material. Japanese Patent HEI 4[1992]-218583, Nishishiro, published August 10, 1992, discloses controlled-release materials including perfumes plus zeolites. U.S. Patent 4,304,675, Corey et al, issued December 8, 1981, teaches a method and composition comprising zeolites for deodorizing articles. East German Patent Publication No. 248,508, published August 12, 1987; East German Patent Publication No. 137,599, published September 12, 1979; European Patent Publication No. 535,942, published April 7, 1993, and Publication No. 536,942, published April 14, 1993, by Unilever PLC; U.S. Patent 5,336,665, issued August 9, 1994 to Garner-Gray et al.; and WO 94/28107, published December 8, 1994.

SUMMARY OF THE INVENTION

The aforementioned needs in the art are met by the present invention which provides a process for producing a particulate laundry additive composition for perfume delivery primarily in laundry detergent and fabric softening products. The process essentially comprises the steps of inputting an aqueous mixture a first encapsulating material and porous carrier particles (e.g., zeolite X) into an extruder, and thereafter, extruding the first encapsulating material, which is preferably a glassy carbohydrate material, with porous carrier particles, preferably loaded with a perfume, so as to form a hot extrudate. Subsequently, the steps of cooling and grinding the extrudate into granules

is complet d. In the final step, a second encapsulating material is coated onto the granules to enrobe further the perfume in the carrier particles. Unexpectedly, as a result of this process, the perfume is sealed significantly better into the carrier material sufficiently so as not to permit exposure or leaching until subjected to the laundering or softening process.

As used herein, the term "extrudate" refers to a continuous phase material formed from an extruder which can have virtually any desired shape. As used herein, the terms "enrobed" or "coated" mean that the carbohydrate material substantially covers the carrier particles regardless of the overall shape of the particles or material to be enrobed or coated, e.g. agglomerates, extrudate or particles. As used herein, the phrase "glass phase" or "glassy" materials refers to microscopically amorphous solid materials having a glass transition temperature, T_g. As used herein, the phrase "continuous phase" refers to a single fused mass of individual or discrete particles. As used herein, the phrase "median particle size" means the "average" particle size in that about 50% of the particles by number (not mass) are larger and about 50% are smaller than this particle size as measured by standard sieve analysis. All percentages and ratios used herein are expressed as percentages by weight (anhydrous basis) unless otherwise indicated. All documents are incorporated herein by reference.

In accordance with one aspect of the invention, a process for producing a particulate laundry additive composition is provided. This process comprises the steps of: (a) inputting a first encapsulating material and porous carrier particles into an extruder, the porous carrier particles having a perfume absorbed therein; (b) extruding the porous carrier particles and the first encapsulating material so as to form an extrudate containing the porous carrier particles enrobed with the first encapsulating material; (c) cooling the extrudate; (d) grinding the extrudate into granules; and (e) coating the granules with a second encapsulating material, thereby forming the particulate laundry additive composition.

In accordance with another aspect of the invention, another process for producing a particulate laundry additive composition is provided. This process comprises the steps of: (a) inputting a first carbohydrate material and porous carrier particles into an extruder, the porous carrier particles having a perfume absorbed therein; (b) mixing the porous carrier particles and the first encapsulating material so as to form a particulate mixture containing the porous carrier particles enrobed with the first encapsulating material; (c) cooling the particulate mixture; (d) grinding the particulate mixture into granules; and (e) coating the granules with a mixture of a second carbohydrate material, thereby forming the particulate laundry additive composition. The present invention also provides th particulate laundry additive composition made according to any one of the processes described herein.

Accordingly, it is an object f the present invention to provide a process for producing a particulate laundry additive composition for perfume delivery in laundry detergent and ther cleaning or fabric softening products. It is also an object of the invention to provide such a process which is more economical, efficient, and one which minimizes product discoloration and the evaporation of perfume and degradation of the materials used during production. These and other objects, features and attendant advantages of the present invention will become apparent to those skilled in the art from a reading of the following detailed description of the preferred embodiment, drawings and the appended claims.

BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is a schematic flow diagram of the process including a preferred embodiment in which the undersized particle recycling step is completed by feeding the undersized particles back to just before the cooling step.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

Process

The process of the invention unexpectedly provides a means by which a perfume-containing particulate laundry additive composition can be prepared without having excessive discoloration and perfume evaporation or degradation during processing and which forms a particulate composition maintaining such perfume prior to its use during the laundering of fabrics. By maintaining the perfume prior to use, it is meant that the perfume is not emitted while stored in the product container, but is only allowed to be emitted during and after deposition on the laundered fabrics as intended. Further, the process unexpectedly prevents the displacement of perfume from the porous carrier particles into the encapsulating material.

While intending not to be bound by theory, it is believed that the process unexpectedly prevents perfume displacement by including a second coating of the encapsulating material onto the perfume-loaded carrier particles. The second coating of encapsulating material is completed subsequent to the sizing operations and may be completed via a variety of known processing techniques. By way of example, the second coating may be sprayed on or completed in a fluid bed coater or similar apparatus. The encapsulating material is preferably a carbohydrate material having a high glass transition temperature (e.g. a Tg greater than 130°C), examples of which are provided hereinafter.

Turning now to Fig. 1 which provides a schematic flow diagram of a preferred embodiment of the process 10, the first step of the process 10 involves inputting a first encapsulating material 6 in aqueous form, and optionally a pigment 8, into a mixer 5 to form an aqueous mixture 7. The mixer 5 can be any conventional tank or vessel having stirring or agitating apparatus included therein. The aqueous mixture 7 is fed to a binder

forming/drying apparatus 12 to form an encapsulating fluid 14. In the binder forming/drying apparatus 12, at least a portion f th water introduced via the aqueous encapsulating material 6 is evaporated via the drying step in this apparatus 12 By a portion, it is meant that the resulting encapsulating fluid 14 contains from about 5% to about 50% of the water originally contained in the encapsulating material 6. Most preferably, however, the encapsulating fluid 14 is substantially free of water.

If the optional pigment 8 is added, it is added in an amount of from about 0.1% to about 10%, and most preferably from about 0.5% to about 5% by weight of the final product. The pigment 8 is preferably selected from the group consisting of titanium dioxide, silica, sodium alumina silicate, ultramarines, optical brighteners and mixtures thereof, although other materials can be used, some of which are listed hereinafter. The most preferred pigment 8 is titanium dioxide. While the pigment 8 is included to prevent discoloration of the ultimate product formed, it may also have the benefit of maintaining the glass transition temperature of the encapsulating fluid 14, lowering its viscosity, and providing superior sealing properties in that the encapsulating fluid is able to prevent emissions of the perfume prior to deposition of the additive on laundered fabrics. The binder/drying apparatus 12 can be a Wiped Film Evaporator (WFE), or heated extruder, in the situation where the encapsulating material 6 is in the molten phase or a conventional spray-drying tower or similar apparatus when the encapsulating material 6 is in the solid phase. Preferably, the encapsulating material 6 is a carbohydrate material, which even more preferably, is in the glass phase after cooling.

In the next step of the process, the encapsulating fluid 14 is inputted to an extruder 16. It should be understood that while extruder 16 can be mixing apparatus, it preferably is an extruder. Porous carrier particles or material 18 as described in detail hereinafter is also added to the extruder 16, preferably near the end of the extruder 16. The extruder 16 can be any known mixing, extrusion, compounding or other apparatus, including but not limited to, extruders commercially available from APV Baker (CP Series), Werner & Pfleiderer (Continua and ZSK Series), Wenger (TF Series); Leistritz (ZSE Series), Buss (LR Series), Reiten Lausar (BT Series); Weber (DS Series), and Columbo (RC Series).

In an optional step of the process invention, a pigment 17 is added to the extruder 16 to aid in the discoloration problem and to modify the viscosity of the mixture being extruded. It should be understood that the benefits of the instant process invention can be achieved by adding the pigment 17 as shown in Fig. 1 and described herein alone, or in addition to the addition of pigment 6 as described previously. The pigment 6 and 17 can be the same, different or various mixtures of the pigment materials described previously. Als , the amount of the pigment 17 added is typically from about 0.1% to about 5%, most preferably from about 1% to about 2% by w ight of the final product. Alternatively, dyes

may be used in place of pigments in a typical amount of from 0.01% to about 1%, m st typically from about 0.02% to about 0.05%.

Preferably, the extruder 16 is maintained at a temperature of from about 50°C t about 200°C, more preferably from about 110°C t about 170°C, and most preferably from about 120°C to about 160°C. In this way, adequate mixing of the porous carrier particles 18 and the encapsulating fluid 14 is ensured. The residence time of the porous carrier particles 18 and the encapsulating fluid 14 in the extruder 16 is preferably from about 0.1 minutes to about 10 minutes, more preferably from about 0.1 minutes to about 5 minutes, and most preferably from about 0.1 minutes to about 2 minutes. Optionally, the extruder 16 can be depressurized to a level of about 100 mm Hg to about 750 mm Hg, more preferably from about 450 mm Hg to about 735 mm Hg, and most preferably from about 710 mm Hg to about 550 mm Hg.

A hot extrudate 20 containing the porous carrier particles 18 enrobed with the encapsulating fluid 14 is formed in the extruder 16 and subjected to a cooling step in preferably a chilled roll/flaker 22 or similar apparatus. The cooling step preferably cools the extrudate 20 to a temperature in a range from about 20°C to about 100°C, more preferably from about 20°C to about 80°C, and most preferably from about 20°C to about 60°C. Preferably, the cooling step is completed within about 1 second to about 120 seconds, more preferably from about 1 second to about 60 seconds, and most preferably from about 1 second to about 30 seconds. The extrudate 20 has a particulate physical form as it exits the chilled roll/flaker 22. In one particular embodiment, the extrudate 20 can be in the form of noodles which are subsequently processed as described hereinafter.

The extrudate 20 are then subjected to a grinding step 24 which can be completed in any know grinding apparatus such as a hammermill. The resulting particles 26 are screened in screening apparatus 28 to provide particles 34 having a median particle size in a range from about 150 microns to about 1100 microns, more preferably from about 200 microns to about 800 microns, and more preferably from about 400 microns to about 600 microns.

Optionally, the process further comprises the step of screening or separating the particles 26 into undersized or "fines" and oversized or "overs" particles, wherein the undersized particles 32 have a median particle size of less than about 150 microns and the oversized particles 30 have a median particle size of at least 1100 microns. In this regard, the aforementioned undersized particles are recycled back to just before the cooling step or chilled roll/flaker 22, while the oversized particles are sent back to the grinding step 24. Past conventional wisdom by the skilled artisan would have recycled the oversized particles 30 and undersized particles 32 back to the extruder 16. However, the recycle steps described herein do not follow this scheme, but rather, recycle back to the cooling and/or

grinding step as appropriate. The use of these process steps results in minimized carbohydrate material and perfume degradation as the recycled particles are only subject to high temperatures for an extremely sh rt period of time.

The particles 34 are subjected to a coating step 36 in which a second encapsulating material 38 is coated onto particles 34 to seal further the perfume into the particles. The encapsulating material 38 can be the same as or different from the encapsulating material 6 as described more fully hereinafter. As alluded to previously, the coating step 36 can be carried forth via conventional spraying techniques or in any standard fluid bed coating apparatus. Additionally, optional plasticizers may be added with encapsulating material 38 which include sorbitol, polyethylene glycol, propylene glycol, low molecular weight carbohydrates and the like. A mixture of encapsulating material 38, sorbitol, and polyethylene glycol is most preferred for this process.

Optionally, mixtures of carbohydrates having various glass transition temperatures can be used in the coating step 36, such carbohydrates are detailed hereinafter and include Capsul E™ and Amiogum 23™ which are commercially available from National Starch Chemical Co. and American Maze Co., respectively. Optionally, a small amount (e.g. 0.01% to 1%) of a surfactant can be included to prevent clumping in the coating apparatus. Further, subsequent drying and cooling steps may be performed to insure that a uniform and smooth coating of the encapsulating material 38 and optional adjunct plasticizers results on the finished particulate laundry additive composition 40.

The laundry additive composition 40 typically comprises from about 10% to about 95% of the encapsulating materials, preferably from about 20% to about 90%, and more preferably from about 20% to about 75% with typical ratios of first encapsulating material to second encapsulating material of about 1:1 to about 10:1, preferably about 5:1 to about 2:1. The additive composition 40 of the present invention also typically comprise from about 0% to about 90% of agents useful for laundry or cleaning compositions, preferably from about 10% to about 80%, and more preferably from about 25% to about 80%.

The laundry additive composition 40 preferably has a hygroscopicity value of less than about 80%. The "hygroscopicity value", as used herein, means the level of moisture uptake by the glassy particles of the composition 40, as measured by the percent increase in weight of the particles under the following test method. The hygroscopicity value required for the present invention glassy particles is determined by placing 2 grams of particles (approximately 500 micron size particles; not having any moisture barrier coating) in an open container petrie dish under conditions of 90°F and 80% relative humidity for a period of 4 weeks. The percent increase in weight of the particles at the end of this time is the particles hygroscopicity value as used herein. Preferred particles

of the composition have a hygroscopicity value f less than about 50%, more preferably less than about 30%.

Particulate Laundry Additive Composition

The process invention produces a particulate laundry additive composition useful in the delivery of perfumes for laundering processes. The composition includes first and second encapsulating materials, 6 and 38, respectively, both of which preferably are a carbohydrate material.

First Encapsulating Material

The first encapsulating material 6 of the present invention is a glassy material derived from one or more at least partially water-soluble hydroxylic compounds. The at least partially water soluble hydroxylic compounds useful herein are preferably selected from the following classes of materials.

1. Carbohydrates, which can be any or a mixture of: i) Simple sugars (or monosaccharides); ii) Oligosaccharides (defined as carbohydrate chains consisting of 2-10 monosaccharide molecules); iii) Polysaccharides (defined as carbohydrate chains consisting of at least 35 monosaccharide molecules); and iv) Starches including modified starches.

Both linear and branched carbohydrate chains may be used. In addition chemically modified starches and poly-/oligo-saccharides may be used. Typical modifications include the addition of hydrophobic moieties of the form of alkyl, aryl, etc. identical to those found in surfactants to impart some surface activity to these compounds.

2. All natural or synthetic gums such as alginate esters, carrageenin, agar-agar, pectic acid, and natural gums such as gum arabic, gum tragacanth and gum karaya.

3. Chitin and chitosan.

4. Cellulose and cellulose derivatives. Examples include: i) Cellulose acetate and Cellulose acetate phthalate (CAP); ii) Hydroxypropyl Methyl Cellulose (HPMC); iii)Carboxymethylcellulose (CMC); iv) all enteric/aquateric coatings and mixtures thereof.

5. Silicates, Phosphates and Borates.

6. Polyvinyl alcohol (PVA).

7. Polyethylene glycol (PEG).

8. Plasticizers.

Materials within these classes which are not at least partially water soluble and which have glass transition temperatures, Tg, below the lower limit herein of about 0°C are useful herein only when mixed in such amounts with the hydroxylic compounds useful herein having the required higher Tg such that the glassy particle produced has the required hygroscopicity value of l ss than about 80%.

Glass transition temperature, commonly abbreviated "Tg", is a well known and readily determined property for glassy materials. This transition is described as being equivalent to the liquification, upon heating through the Tg region, of a material in the glassy state to one in the liquid state. It is not a phase transition such as melting, vaporization, or sublimation. [See William P. Brennan, "'What is a Tg?' A review of the scanning calorimetry of the glass transition", Thermal Analysis Application Study #7, Perkin-Elmer Corporation, March 1973.] Measurement of Tg is readily obtained by using a Differential Scanning Calorimeter.

For purposes of the present invention, the Tg of the hydroxylic compounds is obtained for the anhydrous compound not containing any plasticizer (which will impact the measured Tg value of the hydroxylic compound). Glass transition temperature is also described in detail in P. Peyser, "Glass Transition Temperatures of Polymers", Polymer Handbook, Third Edition, J. Brandrup and E. H. Immergut (Wiley-Interscience; 1989), pp. VI/209 - VI/277.

At least one of the hydroxylic compounds useful in the present invention glassy particles must have an anhydrous, nonplasticized Tg of at least 0°C, and for particles not having a moisture barrier coating, at least about 20 °C, preferably at least about 40°C, more preferably at least 60°C, and most preferably at least about 100°C. It is also preferred that these compounds be low temperature processable, preferably within the range of from about 50°C to about 200°C, and more preferably within the range of from about 60 °C to about 160°C.

Preferably, the hydroxylic compound is a carbohydrate material having a dextrose equivalence, DE, of about 75 or less, more preferably of about 65 or less, most preferably between about 25 and 65, and is a simple sugar. As used herein, the term "dextrose equivalence" and abbreviated "DE", refers to the total amount of reducing sugars expressed as dextrose that is present, calculated as a percentage of the total dry substance. The amount is measured on a scale of 0 to 100 with 100 being the amount present in a pure sugar. The usual technique for determining dextrose equivalence is a volumetric alkaline copper method. Both dextrose equivalence and the methods for measuring dextrose equivalence are well-known in the art particularly in the food and syrup industries. Preferred carbohydrate materials of the first encapsulating material of the present invention include sucrose, glucose, lactose, and corn syrup solids.

Second Encapsulating Material

The second encapsulating material 38 according to the present invention which forms the outer layer is a carbohydrate material having an anhydrous, nonplasticized, glass transition temperature, T_g, of at least about 130°C, and more preferably at least about 150 C, and most preferably about 175°C.

The carbohydrate of the second encapsulating material can be any or a mixture of: i) Simple sugars (or monosaccharides); ii) Oligosaccharides (defined as carbohydrate chains c nsisting of 2-10 monosaccharide molecules); iii) Polysaccharides (defined as carbohydrate chains consisting f at least 35 monosaccharide molecules); and iv) Starches including modified starches.

Both linear and branched carbohydrate chains may be used. In addition chemically modified starches and poly-/oligo-saccharides may be used. Typical modifications include the addition of hydrophobic moieties of the form of alkyl, aryl, etc. identical to those found in surfactants to impart some surface activity to these compounds.

The carbohydrate of the second encapsulating material preferably has a dextrose equivalence, DE, of about 20 or less, more preferably about 15 or less and most preferably about 10 or less. Preferably, the carbohydrate of the second encapsulating material is a starch or modified starch, or maltodextrin. Suitable maltodextrins include Maltrin M040™ commercially available from Grains Products Processing, and suitable starches or modified starches include Capsul E™ and Amiogum 23™ which are commercially available from National Starch Chemical Co. and American Maze Co., respectively.

The second encapsulating material may include optional additive ingredients such as plasticizers, anti-agglomeration agents, and mixtures thereof. The optional plasticizers include sorbitol, polyethylene glycol, propylene glycol, low molecular weight carbohydrates and the like with a mixture of sorbitol and polyethylene glycol with small molecular weight polyols being the most preferred. The plasticizer is employed at levels of from about 0.01% to about 5%. The anti-agglomeration agents according to the present invention are preferably a surfactant and are included at low levels of less than 1% of the second encapsulating material. Suitable surfactants for use in the present invention include TWEEN 80™ commercially available from Imperial Chemicals, Inc..

Porous Carrier Particles

As used herein, "porous carrier particles" means any material capable of supporting (e.g., by adsorption onto the surface or absorption into pores) a perfume agent for incorporation into the particulate compositions. Such materials include porous solids selected from the group consisting of amorphous silicates, crystalline nonlayer silicates, layer silicates, calcium carbonates, calcium/sodium carbonate double salts, sodium carbonates, clays, zeolites, sodalites, alkali metal phosphates, macroporous zeolites, chitin microbeads, carboxyalkylcelluloses, carboxyalkylstarches, cyclodextrins, porous starches and mixtures thereof.

Preferred perfume carrier materials are zeolite X, zeolite Y and mixtures thereof. The term "zeolite" used herein refers to a crystalline aluminosilicate material. The

structural formula of a zeolite is based on the crystal unit cell, the smallest unit of structur represented by

$$Mm/n[(AlO2)m(SiO2)y]\cdot xH2O$$

where n is the valence of the cation M, x is the number of water molecules per unit cell, m and y are the total number of tetrahedra per unit cell, and y/m is 1 to 100. Most preferably, y/m is 1 to 5. The cation M can be Group IA and Group IIA elements, such as sodium, potassium, magnesium, and calcium.

The zeolite useful herein is a faujasite-type zeolite, including Type X Zeolite or Type Y Zeolite, both with a nominal pore size of about 8 Angstrom units, typically in the range of from about 7.4 to about 10 Angstrom units.

The aluminosilicate zeolite materials useful in the practice of this invention are commercially available. Methods for producing X and Y-type zeolites are well- known and available in standard texts. Preferred synthetic crystalline aluminosilicate materials useful herein are available under the designation Type X or Type Y.

For purposes of illustration and not by way of limitation, in a preferred embodiment, the crystalline aluminosilicate material is Type X and is selected from the following:

(I) $Na_{86}[AlO_2]_{86}\cdot(SiO_2)_{106}]\cdot xH_2O$,

(II) $K_{86}[AlO_2]_{86}\cdot(SiO_2)_{106}]\cdot xH_2O$,

(III) $Ca_{40}Na_6[AlO_2]_{86}\cdot(SiO_2)_{106}]\cdot xH_2O$,

(IV) $Sr_{21}Ba_{22}[AlO_2]_{86}\cdot(SiO_2)_{106}]\cdot xH_2O$,

and mixtures thereof, wherein x is from about 0 to about 276. Zeolites of Formula (I) and (II) have a nominal pore size or opening of 8.4 Angstroms units. Zeolites of Formula (III) and (IV) have a nominal pore size or opening of 8.0 Angstroms units.

In another preferred embodiment, the crystalline aluminosilicate material is Type Y and is selected from the following:

(V) $Na_{56}[AlO_2]_{56}\cdot(SiO_2)_{136}]\cdot xH_2O$,

(VI) $K_{56}[AlO_2]_{56}\cdot(SiO_2)_{136}]\cdot xH_2O$

and mixture thereof, wherein x is from about 0 to about 276. Zeolites of Formula (V) and (VI) have a nominal pore size or opening of 8.0 Angstroms units.

Zeolites used in the present invention are in particle form having an average particle size from about 0.5 microns to about 120 microns, preferably from about 0.5 microns to about 30 microns, as measured by standard particle size analysis technique.

The size of the zeolite particles allows them to be entrained in the fabrics with which they come in contact. Once established on the fabric surface (with their coating matrix having been washed away during the laundry process), the zeolites can begin to

release their incorporated laundry agents, especially when subjected to heat or humid conditi ns.

Incorporation of Perfume in Zeolite - The Type X or Type Y Zeolites to be used herein preferably contain less than about 15% desorbable water, m re preferably less than about 8% desorbable water, and most preferably less than about 5% desorbable water. Such materials may be obtained by first activating/dehydrating by heating to about 150 to 350°C, optionally with reduced pressure (from about 0.001 to about 20 Torr). After activation, the agent is slowly and thoroughly mixed with the activated zeolite and, optionally, heated to about 60°C for up to about 2 hours to accelerate absorption equilibrium within the zeolite particles. The perfume/zeolite mixture is then cooled to room temperature and is in the form of a free-flowing powder.

The amount of laundry agent incorporated into the zeolite carrier is less than about 20%, typically less than about 18.5%, by weight of the loaded particle, given the limits on the pore volume of the zeolite. It is to be recognized, however, that the present invention particles may exceed this level of laundry agent by weight of the particle, but recognizing that excess levels of laundry agents will not be incorporated into the zeolite, even if only deliverable agents are used. Therefore, the present invention particles may comprise more than 20% by weight of laundry agents. Since any excess laundry agents (as well as any non-deliverable agents present) are not incorporated into the zeolite pores, these materials are likely to be immediately released to the wash solution upon contact with the aqueous wash medium.

In addition to its function of containing/protecting the perfume in the zeolite particles, the carbohydrate material also conveniently serves to agglomerate multiple perfumed zeolite particles into agglomerates having an overall particles size in the range of 200 to 1000 microns, preferably 400 to 600 microns. This reduces dustiness. Moreover, it lessens the tendency of the smaller, individual perfumed zeolites to sift to the bottom of containers filled with granular detergents, which, themselves, typically have particle sizes in the range of 200 to 1000 microns.

Perfume

As used herein the term "perfume" is used to indicate any odoriferous material which is subsequently released into the aqueous bath and/or onto fabrics contacted therewith. The perfume will most often be liquid at ambient temperatures. A wide variety of chemicals are known for perfume uses, including materials such as aldehydes, ketones, alcohols and esters. More commonly, naturally occurring plant and animal oils and exudates comprising complex mixtures of various chemical components are known for use as perfumes. The perfumes herein can be relatively simple in their compositions or can comprise highly sophisticated complex mixtures of natural and synthetic chemical

components, all chosen to provide any desired odor. Typical perfumes can comprise, for example, woody/earthy bases containing exotic materials such as sandalwood, civet and patchouli oil. The perfumes can be of a light floral fragrance, e.g., rose extract, violet extract, and lilac. The perfumes can also be formulated to provide desirable fruity odors, e.g., lime, lemon, and orange. Any chemically compatible material which exudes a pleasant or otherwise desirable odor can be used in the perfumed compositions herein.

Perfumes also include pro-fragrances such as acetal pro-fragrances, ketal pro-fragrances, ester pro-fragrances (e.g., digeranyl succinate), hydrolyzable inorganic-organic pro-fragrances, and mixtures thereof. These pro-fragrances may release the perfume material as a result of simple hydrolysis, or may be pH-change-triggered pro-fragrances (e.g., pH drop) or may be enzymatically releasable pro-fragrances.

Preferred perfume agents useful herein are defined as follows.

For purposes of the present invention compositions exposed to the aqueous medium of the laundry wash process, several characteristic parameters of perfume molecules are important to identify and define: their longest and widest measures; cross sectional area; molecular volume; and molecular surface area. These values are calculated for individual perfume molecules using the CHEMX program (from Chemical Design, Ltd.) for molecules in a minimum energy conformation as determined by the standard geometry optimized in CHEMX and using standard atomic van der Waal radii. Definitions of the parameters are as follows:

"Longest": the greatest distance (in Angstroms) between atoms in the molecule augmented by their van der Waal radii.

"Widest": the greatest distance (in Angstroms) between atoms in the molecule augmented by their van der Waal radii in the projection of the molecule on a plane perpendicular to the "longest" axis of the molecule.

"Cross Sectional Area": area (in square Angstrom units) filled by the projection of the molecule in the plane perpendicular to the longest axis.

"Molecular Volume": the volume (in cubic Angstrom units) filled by the molecule in its minimum energy configuration.

"Molecular Surface Area": arbitrary units that scale as square Angstroms (for calibration purposes, the molecules methyl beta naphthyl ketone, benzyl salicylate, and camphor gum have surface areas measuring 128 ± 3, 163.5 ± 3, and 122.5 ± 3 units respectively).

The shape of the molecule is also important for incorporation. For example, a symmetric perfectly spherical molecule that is small enough to be included into the zeolite channels has no preferred orientation and is incorporated from any approach direction. However, for molecules that have a length that exceeds th pore dimension,

there is a preferred "approach orientation" for inclusion. Calculation of a molecule's volume/surface area ratio is used h rein to express the "shape index" for a molecule. The higher the value, the more spherical the m lecule.

F r purposes f the present invention, perfume agents are classified according to their ability to be incorporated into zeolite pores, and hence their utility as components for delivery from the zeolite carrier through an aqueous environment. Plotting these agents in a volume/surface area ratio vs. cross sectional area plane permits convenient classification of the agents in groups according to their incorporability into zeolite. In particular, for the zeolite X and Y carriers according to the present invention, agents are incorporated if they fall below the line (herein referred to as the "incorporation line") defined by the equation:

$$y = -0.01068x + 1.497$$

where x is cross sectional area and y is volume/surface area ratio. Agents that fall below the incorporation line are referred to herein as "deliverable agents"; those agents that fall above the line are referred to herein as "non-deliverable agents".

For containment through the wash, deliverable agents are retained in the zeolite carrier as a function of their affinity for the carrier relative to competing deliverable agents. Affinity is impacted by the molecule's size, hydrophibicity, functionality, volatility, etc., and can be effected via interaction between deliverable agents within the zeolite carrier. These interactions permit improved through the wash containment for the deliverable agents mixture incorporated. Specifically, for the present invention, the use of deliverable agents having at least one dimension that is closely matched to the zeolite carrier pore dimension slows the loss of other deliverable agents in the aqueous wash environment. Deliverable agents that function in this manner are referred to herein as "blocker agents", and are defined herein in the volume/surface area ratio vs. cross sectional area plane as those deliverable agent molecules falling below the "incorporation line" (as defined herein before) but above the line (herein referred to as the "blocker line") defined by the equation:

$$y = -0.01325x + 1.46$$

where x is cross sectional area and y is volume/surface area ratio.

For the present invention compositions which utilize zeolite X and Y as the carriers, all deliverable agents below the "incorporation line" can be delivered and released from the present invention compositions, with the preferred materials being those falling below the "blocker line". Also preferred are mixtures of blocker agents and other deliverable agents. Laundry perfume agent mixtures useful for the present invention laundry particles preferably comprise from about 5% to about 100% (preferably from about 25% to about 100%; more preferably from about 50% to about

100%) deliverable agents; and preferably comprising from about 0.1% to about 100% (preferably from about 0.1% to about 50%) blocker agents, by weight of the laundry agents mixture.

Obviously for the present invention compositions whereby perfume agents are being delivered by the compositions, sensory perception is required for a benefit to be seen by the consumer. For the present invention perfume compositions, the most preferred perfume agents useful herein have a threshold of noticability (measured as odor detection thresholds ("ODT") under carefully controlled GC conditions as described in detail hereinafter) less than or equal to 10 parts per billion ("ppb"). Agents with ODTs between 10 ppb and 1 part per million ("ppm") are less preferred. Agents with ODTs above 1 ppm are preferably avoided. Laundry agent perfume mixtures useful for the present invention laundry particles preferably comprise from about 0% to about 80% of deliverable agents with ODTs between 10 ppb and 1 ppm, and from about 20% to about 100% (preferably from about 30% to about 100%; more preferably from about 50% to about 100%) of deliverable agents with ODTs less than or equal to 10 ppb.

Also preferred are perfumes carried through the laundry process and thereafter released into the air around the dried fabrics (e.g., such as the space around the fabric during storage). This requires movement of the perfume out of the zeolite pores with subsequent partitioning into the air around the fabric. Preferred perfume agents are therefore further identified on the basis of their volatility. Boiling point is used herein as a measure of volatility and preferred materials have a boiling point less than 300°C. Laundry agent perfume mixtures useful for the present invention laundry particles preferably comprise at least about 50% of deliverable agents with boiling point less than 300°C (preferably at least about 60%; more preferably at least about 70%).

In addition, preferred laundry particles herein comprise compositions wherein at least about 80%, and more preferably at least about 90%, of the deliverable agents have a "ClogP value" greater than about 1.0. ClogP values are obtained as follows.

Calculation of ClogP:

These perfume ingredients are characterized by their octanol/water partition coefficient P. The octanol/water partition coefficient of a perfume ingredient is the ratio between its equilibrium concentration in octanol and in water. Since the partition coefficients of most perfume ingredients are large, they are more conveniently given in the form of their logarithm to the base 10, logP.

The logP of many perfume ingredients has been reported; for example, the Pomona92 database, available from Daylight Chemical Information Systems, Inc. (Daylight CIS), contains many, along with citations to the original literature.

However, the l gP values are most conveniently calculated by the "CLOGP" program, also available from Daylight CIS. This program also lists experimental logP values when they are available in the Pomona92 database. The "calculated logP" (ClogP) is determined by the fragm nt approach of Hansch and Leo (cf., A. Leo, in Comprehensive Medicinal Chemistry, Vol. 4, C. Hansch, P.G. Sammens, J. B. Taylor and C. A. Ramsden, Eds., p. 295, Pergamon Press, 1990). The fragment approach is based on the chemical structure of each perfume ingredient and takes into account the numbers and types of atoms, the atom connectivity, and chemical bonding. The ClogP values, which are the most reliable and widely used estimates for this physicochemical property, can be used instead of the experimental logP values in the selection of perfume ingredients.

Determination of Odor Detection Thresholds:

The gas chromatograph is characterized to determine the exact volume of material injected by the syringe, the precise split ratio, and the hydrocarbon response using a hydrocarbon standard of known concentration and chain-length distribution. The air flow rate is accurately measured and, assuming the duration of a human inhalation to last 0.2 minutes, the sampled volume is calculated. Since the precise concentration at the detector at any point in time is known, the mass per volume inhaled is known and hence the concentration of material. To determine whether a material has a threshold below 10 ppb, solutions are delivered to the sniff port at the back-calculated concentration. A panelist sniffs the GC effluent and identifies the retention time when odor is noticed. The average over all panelists determines the threshold of noticeability.

The necessary amount of analyte is injected onto the column to achieve a 10 ppb concentration at the detector. Typical gas chromatograph parameters for determining odor detection thresholds are listed below.

GC: 5890 Series II with FID detector

7673 Autosampler

Column: J&W Scientific DB-1

Length 30 meters ID 0.25 mm film thickness 1 micron

Method:

Split Injection: 17/1 split ratio

Autosampler: 1.13 microliters per injection

Column Flow: 1.10 mL/minute

Air Flow: 345 mL/minute

Inlet Temp. 245°C

Detector Temp. 285°C

Temperature Information

Initial Temperature: 50°C

Rate: 5C/minute

Final Temperature: 280°C

Final Time: 6 minutes

Leading assumptions: 0.02 minutes per sniff

GC air adds to sample dilution

Perfume Fixative:

Optionally, the perfume can be combined with a perfume fixative. The perfume fixative materials employed herein are characterized by several criteria which make them especially suitable in the practice of this invention. Dispersible, toxicologically-acceptable, non-skin irritating, inert to the perfume, degradable and/or available from renewable resources, and relatively odorless additives are used. Perfume fixatives are believed to slow the evaporation of more volatile components of the perfume.

Examples of suitable fixatives include members selected from the group consisting of diethyl phthalate, musks, and mixtures thereof. If used, the perfume fixative comprises from about 10% to abut 50%, preferably from about 20% to about 40%, by weight, of the perfume.

Adjunct Laundry or Cleaning Ingredients

Adjunct ingredients useful for in or with the laundry or cleaning particulate compositions according to the present invention are selected from the group consisting of surfactants, perfumes, bleaches, bleach promoters, bleach activators, bleach catalysts, chelants, antiscalants, threshold inhibitors, dye transfer inhibitors, photobleaches, enzymes, catalytic antibodies, brighteners, fabric-substantive dyes, antifungals, antimicrobials, insect repellents, soil release polymers, fabric softening agents, dye fixatives, pH jump systems, and mixtures thereof. As can be appreciated for the present invention, these agents useful for laundry or cleaning compositions which are incorporated into the particulate compositions of the present invention may be the same as or different from those agents which are used to formulate the remainder of the laundry and cleaning compositions containing the particulate compositions produced by the instant process. For example, the particulate compositions may comprise a perfume agent and the same or different agent may also be blended into the final composition along with the perfume-containing particulate composition. These agents are selected as desired for the type of composition being formulated, such as granular laundry detergent compositions, granular automatic dishwashing compositions, or hard surface cleaners.

The various types of agents useful in laundry and cleaning compositions are described hereinafter. The compositions containing particulate compositions can optionally include one or more oth r deterg nt adjunct materials or other materials for

assisting r enhancing cleaning performance, treatment of the substrate to be cleaned, or to modify the aesthetics of the detergent composition.

Detersive Surfactant

The granules and/or the agglomerates include surfactants at the levels stated previously. The detersive surfactant can be selected from the group consisting of anionic surfactants, nonionic surfactants, cationic surfactants, zwitterionic surfactants and mixtures. Nonlimiting examples of surfactants useful herein include the conventional C_{11}-C_{18} alkyl benzene sulfonates ("LAS") and primary, branched-chain and random C_{10}-C_{20} alkyl sulfates ("AS"), the C_{10}-C_{18} secondary (2,3) alkyl sulfates of the formula $CH_3(CH_2)_x(CHOSO_3^-M^+)CH_3$ and $CH_3(CH_2)_y(CHOSO_3^-M^+)CH_2CH_3$ where x and (y + 1) are integers of at least about 7, preferably at least about 9, and M is a water-solubilizing cation, especially sodium, unsaturated sulfates such as oleyl sulfate, the C_{10}-C_{18} alkyl alkoxy sulfates ("AE_xS"; especially EO 1-7 ethoxy sulfates), C_{10}-C_{18} alkyl alkoxy carboxylates (especially the EO 1-5 ethoxycarboxylates), the C_{10-18} glycerol ethers, the C_{10}-C_{18} alkyl polyglycosides and their corresponding sulfated polyglycosides, and C_{12}-C_{18} alpha-sulfonated fatty acid esters. If desired, the conventional nonionic and amphoteric surfactants such as the C_{12}-C_{18} alkyl ethoxylates ("AE") including the so-called narrow peaked alkyl ethoxylates and C_6-C_{12} alkyl phenol alkoxylates (especially ethoxylates and mixed ethoxy/propoxy), C_{12}-C_{18} betaines and sulfobetaines ("sultaines"), C_{10}-C_{18} amine oxides, and the like, can also be included in the overall compositions. The C_{10}-C_{18} N-alkyl polyhydroxy fatty acid amides can also be used. Typical examples include the C_{12}-C_{18} N-methylglucamides. See WO 9,206,154. Other sugar-derived surfactants include the N-alkoxy polyhydroxy fatty acid amides, such as C_{10}-C_{18} N-(3-methoxypropyl) glucamide. The N-propyl through N-hexyl C_{12}-C_{18} glucamides can be used for low sudsing. C_{10}-C_{20} conventional soaps may also be used. If high sudsing is desired, the branched-chain C_{10}-C_{16} soaps may be used. Mixtures of anionic and nonionic surfactants are especially useful. Other conventional useful surfactants are listed in standard texts.

The C_{10}-C_{18} alkyl alkoxy sulfates ("AE_xS"; especially EO 1-7 ethoxy sulfates) and C_{12}-C_{18} alkyl ethoxylates ("AE") are the most preferred for the cellulase-containing detergents described herein.

Detersive Builder

The granules and agglomerates preferably include a builder at the previously stated levels. To that end, inorganic as well as organic builders can be used. Also, crystalline as well as amorphous builder materials can be used. Builders are typically used in fabric laundering compositions to assist in the removal of particulate soils and to eliminate or minimize the effects of water hardness during eashing processes.

Inorganic or P-containing detergent builders include, but are not limited to, the alkali metal, ammonium and alkanolammonium salts of polyphosphates (exemplified by the tripolyphosphates, pyrophosphates, and glassy polymeric meta-ph sphates), phosphonates, phytic acid, silicates, carbonates (including bicarbonates and sesquicarbonates), sulphates, and aluminosilicates. However, non-phosphate builders are required in some locales. Importantly, the compositions herein function surprisingly well even in the presence of the so-called "weak" builders (as compared with phosphates) such as citrate, or in the so-called "under built" situation that may occur with zeolite or layered silicate builders.

Examples of silicate builders are the alkali metal silicates, particularly those having a SiO_2:Na_2O ratio in the range 1.6:1 to 3.2:1 and layered silicates, such as the layered sodium silicates described in U.S. Patent 4,664,839, issued May 12, 1987 to H. P. Rieck. NaSKS-6 is the trademark for a crystalline layered silicate marketed by Hoechst (commonly abbreviated herein as "SKS-6"). Unlike zeolite builders, the Na SKS-6 silicate builder does not contain aluminum. NaSKS-6 has the delta-Na_2SiO_5 morphology form of layered silicate. It can be prepared by methods such as those described in German DE-A-3,417,649 and DE-A-3,742,043. SKS-6 is a highly preferred layered silicate for use herein, but other such layered silicates, such as those having the general formula $NaMSi_xO_{2x+1} \cdot yH_2O$ wherein M is sodium or hydrogen, x is a number from 1.9 to 4, preferably 2, and y is a number from 0 to 20, preferably 0 can be used herein. Various other layered silicates from Hoechst include NaSKS-5, NaSKS-7 and NaSKS-11, as the alpha, beta and gamma forms. As noted above, the delta-Na_2SiO_5 (NaSKS-6 form) is most preferred for use herein. Other silicates may also be useful such as for example magnesium silicate, which can serve as a crispening agent in granular formulations, as a stabilizing agent for oxygen bleaches, and as a component of suds control systems.

Examples of carbonate builders are the alkaline earth and alkali metal carbonates as disclosed in German Patent Application No. 2,321,001 published on November 15, 1973. As mentioned previously, aluminosilicate builders are useful builders in the present invention. Aluminosilicate builders are of great importance in most currently marketed heavy duty granular detergent compositions, and can also be a significant builder ingredient in liquid detergent formulations. Aluminosilicate builders include those having the empirical formula:

$$M_z(zAlO_2)_y] \cdot xH_2O$$

wherein z and y are integers of at least 6, the molar ratio of z to y is in the range from 1.0 to about 0.5, and x is an integer from about 15 to about 264.

Useful aluminosilicate i n exchange materials are commercially available. These aluminosilicates can be crystalline or amorphous in structur and can be naturally-

occurring aluminosilicates or synthetically derived. A method for producing aluminosilicate ion exchange materials is disclosed in U.S. Patent 3,985,669, Krummel, et al, issued Oct ber 12, 1976. Preferred synthetic crystalline aluminosilicate ion exchange materials useful herein are available under the designations Zeolite A, Zeolite P (B), Zeolite MAP and Zeolite X. In an especially preferred embodiment, the crystalline aluminosilicate ion exchange material has the formula:

$$Na_{12}[(AlO_2)_{12}(SiO_2)_{12}]\cdot xH_2O$$

wherein x is from about 20 to about 30, especially about 27. This material is known as Zeolite A. Dehydrated zeolites (x = 0 - 10) may also be used herein. Preferably, the aluminosilicate has a particle size of about 0.1-10 microns in diameter.

Organic detergent builders suitable for the purposes of the present invention include, but are not restricted to, a wide variety of polycarboxylate compounds. As used herein, "polycarboxylate" refers to compounds having a plurality of carboxylate groups, preferably at least 3 carboxylates. Polycarboxylate builder can generally be added to the composition in acid form, but can also be added in the form of a neutralized salt. When utilized in salt form, alkali metals, such as sodium, potassium, and lithium, or alkanolammonium salts are preferred.

Included among the polycarboxylate builders are a variety of categories of useful materials. One important category of polycarboxylate builders encompasses the ether polycarboxylates, including oxydisuccinate, as disclosed in Berg, U.S. Patent 3,128,287, issued April 7, 1964, and Lamberti et al, U.S. Patent 3,635,830, issued January 18, 1972. See also "TMS/TDS" builders of U.S. Patent 4,663,071, issued to Bush et al, on May 5, 1987. Suitable ether polycarboxylates also include cyclic compounds, particularly alicyclic compounds, such as those described in U.S. Patents 3,923,679; 3,835,163; 4,158,635; 4,120,874 and 4,102,903.

Other useful detergency builders include the ether hydroxypolycarboxylates, copolymers of maleic anhydride with ethylene or vinyl methyl ether, 1, 3, 5-trihydroxy benzene-2, 4, 6-trisulphonic acid, and carboxymethyloxysuccinic acid, the various alkali metal, ammonium and substituted ammonium salts of polyacetic acids such as ethylenediamine tetraacetic acid and nitrilotriacetic acid, as well as polycarboxylates such as mellitic acid, succinic acid, oxydisuccinic acid, polymaleic acid, benzene 1,3,5-tricarboxylic acid, carboxymethyloxysuccinic acid, and soluble salts thereof.

Citrate builders, e.g., citric acid and soluble salts thereof (particularly sodium salt), are polycarboxylate builders of particular importance for heavy duty liquid detergent formulations due to their availability from renewable resources and their biodegradability. Citrates can also be used in granular compositions, especially in combination with zeolite

and/ r layered silicate builders. Oxydisuccinates are also especially useful in such compositions and combinations.

Also suitable in the detergent compositions of the present invention are the 3,3-dicarboxy-4-oxa-1,6-hexanedioates and the related compounds disclosed in U.S. Patent 4,566,984, Bush, issued January 28, 1986. Useful succinic acid builders include the C_5-C_{20} alkyl and alkenyl succinic acids and salts thereof. A particularly preferred compound of this type is dodecenylsuccinic acid. Specific examples of succinate builders include: laurylsuccinate, myristylsuccinate, palmitylsuccinate, 2-dodecenylsuccinate (preferred), 2-pentadecenylsuccinate, and the like. Laurylsuccinates are the preferred builders of this group, and are described in European Patent Application 86200690.5/0,200,263, published November 5, 1986.

Other suitable polycarboxylates are disclosed in U.S. Patent 4,144,226, Crutchfield et al, issued March 13, 1979 and in U.S. Patent 3,308,067, Diehl, issued March 7, 1967. See also Diehl U.S. Patent 3,723,322.

Fatty acids, e.g., C_{12}-C_{18} monocarboxylic acids, can also be incorporated into the compositions alone, or in combination with the aforesaid builders, especially citrate and/or the succinate builders, to provide additional builder activity. Such use of fatty acids will generally result in a diminution of sudsing, which should be taken into account by the formulator.

In situations where phosphorus-based builders can be used, and especially in the formulation of bars used for hand-laundering operations, the various alkali metal phosphates such as the well-known sodium tripolyphosphates, sodium pyrophosphate and sodium orthophosphate can be used. Phosphonate builders such as ethane-1-hydroxy-1,1-diphosphonate and other known phosphonates (see, for example, U.S. Patents 3,159,581; 3,213,030; 3,422,021; 3,400,148 and 3,422,137) can also be used.

Other Adjunct Ingredients

The detergent composition may also include enzymes, enzyme stabilizers, brighteners, polymeric dispersing agents (i.e. polyacrylates), carriers, hydrotropes, suds boosters or suppressers, soil release agents, dye transfer inhibitors, and processing aids.

In order to make the present invention more readily understood, reference is made to the following examples, which are intended to be illustrative only and not intended to be limiting in scope.

Example I

A solution of 82% solid carbohydrate material (having a dextrose equivalence 62) and the balance water is premixed in an agitated mixing vessel with 1.5% by weight TiO_2 powder (comm rcially sold under the trade name Tronox by the Kerr McGee Chemical Corporation) to form a carbohydrate encapsulation fluid solution. The carbohydrate fluid is

dried to a m isture level of 2% in a Luwa™ Wiped Film Evaporator ("WFE") and is then cooled to form a solid glassy material. The resulting carbohydrate fluid has a moisture level of 2.0%. Thereafter, the carbohydrate fluid and zeolite X loaded with 16% perfume by weight ("PLZ") are inputted at a weight ratio of 1:1 into a 12 barrel, Werner & Pfleiderer™ ZSK 30 twin screw extruder ("TSE") without a constricting die plate to form agglomerates. Barrels 1 through 4 of the TSE are maintained at a temperature of 80°C while barrels 5 and 6 are maintained at a temperature of 90°C, barrels 7 and 8 at a temperature of 130°C, barrels 9 and 10 at a temperature of 135°C, and barrels 11 and 12 at a temperature of 13°C. The carbohydrate fluid is fed at a temperature of 160°C to the TSE in barrel 7, while the PLZ is added in barrel 11 and intimately mixed with the carbohydrate fluid prior to leaving the TSE as an extrudate having a discharge temperature of 145°C and a rate of 500 g/min. The product is cooled at room temperature to form free flowing particles which are ground in a Fitz Mill™ (commercially available from the Fitzpatrick Company) and sized via screening to result in particles in the size range of 150 microns to 1180 microns. The sized particles are then sent to a Wurster fluid bed coater in which an aqueous mixture containing 22.5% of Maltrin M040™ commercially available from Grain Processing Corporation, 1.0% of D-Sorbitol™ commercially available from J.T. Baker, 1.0% of polyethylene glycol (Carbowax™ PEG 600 commercially available from Union Carbide), and 0.5% of surfactant (TWEEN 80™ commercially available from Imperial Chemicals, Inc.(ICI)) is added. The coated particles are dried to produce a particulate composition extremely suitable for use as a laundry additive composition.

The particles formed unexpectedly have a superior "Neat Product Odor" ("NPO") in that it does not emit any detectable odor over the base product odor as observed by a statistically significant number of panelist graders. This provides strong evidence of the lack of perfume displacement from the carrier particles.

Example II

Several detergent compositions made in accordance with the invention and specifically for top-loading washing machines are exemplified below incorporating the perfume particle prepared in Example I.

	% Weight		
Base Granule	**A**	**B**	**C**
Aluminosilicate	18.0	22.0	24.0
Sodium Sulfate	10.0	19.0	6
Sodium Polyacrylate Polymer	3.0	2.0	4.0
Polyethylene Glycol (MW=400)	2.0	1.0	--
C_{12-13} Linear Alkylbenzene Sulfonate, Na	6.0	7.0	8.0
C_{14-16} Secondary Alkyl Sulfate, Na	3.0	3.0	--

C_{14-15} Alkyl Ethoxylated Sulfate, Na	3.0	9.0	--
Sodium Silicate	1.0	2.0	3.0
Brightener 24/47[1]	0.3	0.3	0.3
Sodium Carbonate	7.0	26.0	
Carboxymethyl Cellulose	--	--	1.0
DTPMPA[2]	--	--	0.5
DTPA[3]	0.5	--	--
Admixed Agglomerates			
C_{14-15} Alkyl Sulfate, Na	5.0	--	--
C_{12-13} Linear Alkylbenzene Sulfonate, Na	2.0	--	--
Sodium Carbonate	4.0	--	--
Polyethylene Glycol (MW=4000)	1.0	--	--
Admix			
Sodium Carbonate	--	--	13.0
C_{12-15} Alkyl Ethoxylate (EO=7)	2.0	0.5	2.0
C_{12-15} Alkyl Ethoxylate (EO=3)	--	--	2.0
Perfume Spray-On	0.3	0.4	0.3
Perfume Particles[4]	0.5	0.5	0.5
Polyvinylpyrrilidone	0.5	--	--
Polyvinylpyridine N-oxide	0.5	--	--
Polyvinylpyrrolidone-polyvinylimidazole	0.5	--	--
Distearylamine & Cumene Sulfonic Acid	2.0	--	--
Soil Release Polymer[5]	0.5	--	--
Lipolase Lipase (100,000 LU/I)[6]	0.5	--	0.5
Termamyl Amylase (60 KNU/g)[6]	0.3	--	0.3
CAREZYME® Cellulase (1000 CEVU/g)[6]	0.3	--	--
Protease (40mg/g)[7]	0.5	0.5	0.5
NOBS[8]	5.0	--	--
TAED[9]	--	--	3.0
Sodium Percarbonate	12.0	--	--
Sodium Perborate Monohydrate	--	--	22.0
Polydimethylsiloxane	0.3	--	3.0
Sodium Sulfate	--	--	3.0
Miscellaneous (water, etc.)	balance	balance	balance
Total	100.0	100.0	100.0

[1] Purchased from Ciba-Geigy

[2] Diethyl ne Triamine Pentamethylene Phosophonic Acid

[3] Diethylene Triamine Pentaacetic Acid

[4] From Example I

[5] Made according to U.S. Patent 5,415,807, issued May 16, 1995 to Gosselink et al

[6] Purchased from Novo Nordisk A/S

[7] Purchased from Genencor

[8] Nonanoyloxybenzenesulfonate

[9] Tetra Acetyle Ethylene Dramine

Example III

The following detergent compositions containing a perfume particle from Example I in accordance with the invention are especially suitable for front loading washing machines.

	% Weight	
Base Granule	**A**	**B**
Aluminosilicate	15.0	--
Sodium Sulfate	2.0	--
C_{12-13} Linear Alkylbenzene Sulfonate, Na	3.0	--
DTPMPA[1]	0.5	--
Carboxymethylcellulose	0.5	--
Acrylic Acid/Maleic Acid Co-polymer	4.0	--
Admixed Agglomerates		
C_{14-15} Alkyl Sulfate, Na	--	11.0
C_{12-13} Linear Alkylbenzene Sulfonate, Na	5.0	--
C_{18-22} Alkyl Sulfate, Na	2.0	--
Sodium Silicate	4.0	--
Aluminosilicate	12.0	13.0
Carboxymethylcellulose	--	0.5
Acrylic Acid/Maleic Acid Co-polymer	--	2.0
Sodium Carbonate	8.0	7.0
Admix		
Perfume Spray-On	0.3	0.5
Perfume Particles[2]	0.5	0.5
C_{12-15} Alkyl Ethoxylate (EO=7)	4.0	4.0
C_{12-15} Alkyl Ethoxylate (EO=3)	2.0	2.0
Acrylic Acid/Maleic Acid Co-polymer	--	3.0
Crystalline Layered Silicate[3]	--	12.0
Sodium Citrate	5.0	8.0
Sodium Bicarbonate	5.0	5.0

Sodium Carbonate	6.0	15.0
Polyvinylpyrrilidone	0.5	0.5
Alcalase protease[4] (3.0 AU/g)	0.5	1.0
Lipolase Lipase[4] (100,000 LU/l)	0.5	0.5
Termamyl Amylase[4] (60KNU/g)	0.5	0.5
CAREZYME® Cellulase[4] (1000CEVU/g)	0.5	0.5
Sodium Sulfate	4.0	0.0
Miscellaneous (water, etc.)	balance	balance
Total	100.0	100.0

[1] Diethylene Triamine Pentamethylenephosphonic Acid

[2] From Example I

[3] SKS 6 commercially available from Hoechst

[4] Purchased from Novo Nordisk A/S

Example IV

The following detergent compositions according to the invention are suitable for low wash volume, top loading washing machines.

	(% Weight)
Base Granules	A
Aluminosilicate	7.0
Sodium Sulfate	3.0
Polyethylene Glycol (MW=4000)	0.5
Acrylic Acid/Maleic Acid Co-polymer	6.0
Cationic Surfactant[1]	0.5
C_{14-16} Secondary Alkyl Sulfate, Na	7.0
C_{12-13} Linear Alkylbenzene Sulfonate, Na	13.0
C_{14-15} Alkyl Ethoxylated Sulfate, Na	6.0
Crystalline Layered Silicate[2]	6.0
Sodium Silicate	2.0
Oleic Fatty Acid, Na	1.0
Brightener 49[3]	0.3
Sodium Carbonate	28.0
DTPA[4]	0.3
Admix	
C_{12-15} Alkyl Ethoxylate (EO=7)	1.0
Perfume Spray-On	1.0
Perfume Particles[5]	1.0
Soil Release Polymer[6]	0.5
Polyvinylpyrrilidone	0.3
Polyvinylpyridine N-Oxide	0.1
Polyvinylpyrrilidone-polyvinylimidazole	0.1
Lipolase Lipase (100.000LU/g)[7]	0.3
Termamyl Amylase (60KNU/g)[7]	0.1
CAREZYME® Cellulase (1000 CEVU/g)[7]	0.1
Savinase (4.0 KNPU/g)[7]	1.0
NOBS[8]	4.0
Sodium Perborate Monohydrate	5.0
Miscellaneous (water, etc.)	balance
Total	100.0

[1] C_{12-14} Dimethyl Hydroxyethyl Quaternary Ammonium Compound

[2] SKS 6 commercially available from Hoechst

[3] Purchased from Ciba-Geigy

[4] Diethylene Triamine Pentaacetic Acid

[5] From Example I

[6] Made according to U.S. patent 5,415,807 issued May 16, 1995 to Gosselink et al

[7] Purchased from Novo Nordisk A/S

[8] Nonanoyloxybenzenesulfonate

Example V

The following detergent compositions according to the invention are suitable for machine and handwashing operations. The base granule is prepared by a conventional spray drying process in which the starting ingredients are formed into a slurry and passed through a spray drying tower having a counter current stream of hot air (200-400°C) resulting in the formation of porous granules. The remaining adjunct detergent ingredients are sprayed on or added dry.

	(% Weight)		
Base Granule	**A**	**B**	**C**
C_{12-13} Alkylbenzene Sulfonate, Na	19.0	18.0	19.0
Cationic Surfactant[1]	0.5	0.5	--
DTPMPA[2]	0.3	--	--
DTPA[3]	--	0.3	--
Sodium Tripolyphosphate	25.0	19.0	29.0
Acrylic/Maleic Co-polymer	1.0	0.6	--
Carboxymethylcellulose	0.3	0.2	0.3
Brightener 49/15/33[4]	0.2	0.2	0.2
Sodium Sulfate	28.0	39.0	15.0
Sodium Silicate (2.0R)	7.5	--	--
Sodium Silicate (1.6R)	--	7.5	6.0
Admix			
Sodium Carbonate	5.0	6.0	20.0
C_{12-13} Alkyl Ethoxylate (EO=7)	0.4	--	1.2
Savinase[5] Protease (4KNPY/g)	0.6	--	1.0
Termamyl[5] Amylase (60KNU/g)	0.4	--	--
Lipolase[5] Lipase (100,000 LU/I)	0.1	0.1	0.1
Sav/Ban[5] (6 KNPU/100 KNU/g)	--	0.3	--
CAREZYME®[5] Cellulase (1000 CEVU/g)	--	0.1	--
Soil Release Polymer[6]	0.1	0.1	0.3
Perfume Spray-On	0.4	0.4	0.4
Quantum(zinc phthalocyanine sulfonate)	2.0	2.0	2.0

Perfume Particles[7]	1.5	1.5	2.0
Miscellaneous (water, etc.)	balance	balance	balance
Total	100.0	100.0	100.0

[1] C_{12-14} Dimethyl Hydroxyethyl Quaternary Ammonium Compound

[2] Diethylene Triamine Pentamethylenephosphoric Acid

[3] Diethylene Triamine Pentaacetic Acid

[4] Purchased from Ciba-Geigy

[5] Purchased from Novo Nordisk A/S

[6] Made according to U.S. patent 5,415,807 issued May 16, 1995 to Gosselink et al

[7] From Example I

Example VI

The following detergent composition according to the invention is in the form of a laundry bar which is particularly suitable for handwashing operations.

	(% Weight)
	A
Coconut Fatty Alkyl Sulfate	30.0
Sodium Tripolyphosphate	5.0
Tetrasodium Pyrophosphate	5.0
Sodium Carbonate	20.0
Sodium Sulfate	5.0
Calcium Carbonate	5.0
$Na_{1.9}K_{0.1}Ca(CO_3)_2$	15.0
Aluminosilicate	2.0
Coconut Fatty Alcohol	2.0
Perfume Particles[1]	1.0
Perfume Spray-On	1.0
Miscellaneous (water, etc.)	balance
Total	100.0

[1] From Example I.

Having thus described the invention in detail, it will be clear to those skilled in the art that various changes may be made without departing from the scope of the invention and the invention is not to be considered limited to what is described in the specification.

WHAT IS CLAIMED IS:

1. A process for producing a particulate laundry additive composition characterized by the steps of:

(a) inputting a first encapsulating material and porous carrier particles into an extruder, said porous carrier particles having a perfume absorbed therein;

(b) extruding said porous carrier particles and said first encapsulating material so as to form an extrudate containing said porous carrier particles enrobed with said first encapsulating material;

(c) cooling said extrudate;

(d) grinding said extrudate into granules; and

(e) coating said granules with a second encapsulating material, thereby forming said particulate laundry additive composition.

2. The process according to claim 1 wherein said first and second encapsulating materials are substantially free of water.

3. The process according to claims 1-2 wherein said first and second encapsulating materials are in the glass phase and have a glass transition temperature in the range of from 30°C to 200°C.

4. The process according to claims 1-3 wherein said first and second encapsulating materials are selected from starches, polysaccharides, oligosaccharides, disaccharides, monosaccharidesalginate esters, carrageenin, agar-agar, pectic acid, chitosan, chitin, cellulose acetate cellulose acetate phthalate, carboxymethylcellulase, silicates, phosphates, borates, polyethylene glycols, polyvinyl alcohol, nonionic surfactants and mixtures thereof.

5. The process according to claims 1-4 further characterized by the step of adding a pigment to said extruder.

6. The process according to claim 5 wherein said pigment is selected from the group consisting of titanium dioxide, silica, sodium alumina silicate, ultramarines, optical brighteners and mixtures thereof.

7. The process according to claims 1-6 wherein said cooling step includes cooling said extrudate to be within a temperature range of from 20°C to 100°C.

8. The process according to claims 1-7 wherein said cooling step is completed within 1 second to 120 seconds.

9. The process according to claims 1-8 wherein said porous carrier material is selected from the group consisting of amorphous silicates, crystalline nonlayered silicates, layered silicates, calcium carbonates, calcium/sodium carbonate double salts, sodium carbonates, clays, zeolites, sodalites, alkali metal phosphates, macroporous zeolites, chitin microbeads, carboxyalkylcelluloses, carboxyalkylstarches, cyclodextrins, porous starches and mixtures thereof; and said porous solid has a surface area of at least 50 m^2/g.

10. A process for producing a particulate laundry additive composition characterized by the steps of:

(a) inputting a first carbohydrate material and porous carrier particles into an extruder, said porous carrier particles having a perfume absorbed therein;
(b) mixing said porous carrier particles and said first encapsulating material so as to form a particulate mixture containing said porous carrier particles enrobed with said first encapsulating material;
(c) cooling said particulate mixture;
(d) grinding said particulate mixture into granules; and
(e) coating said granules with a second carbohydrate material, thereby forming said particulate laundry additive composition.

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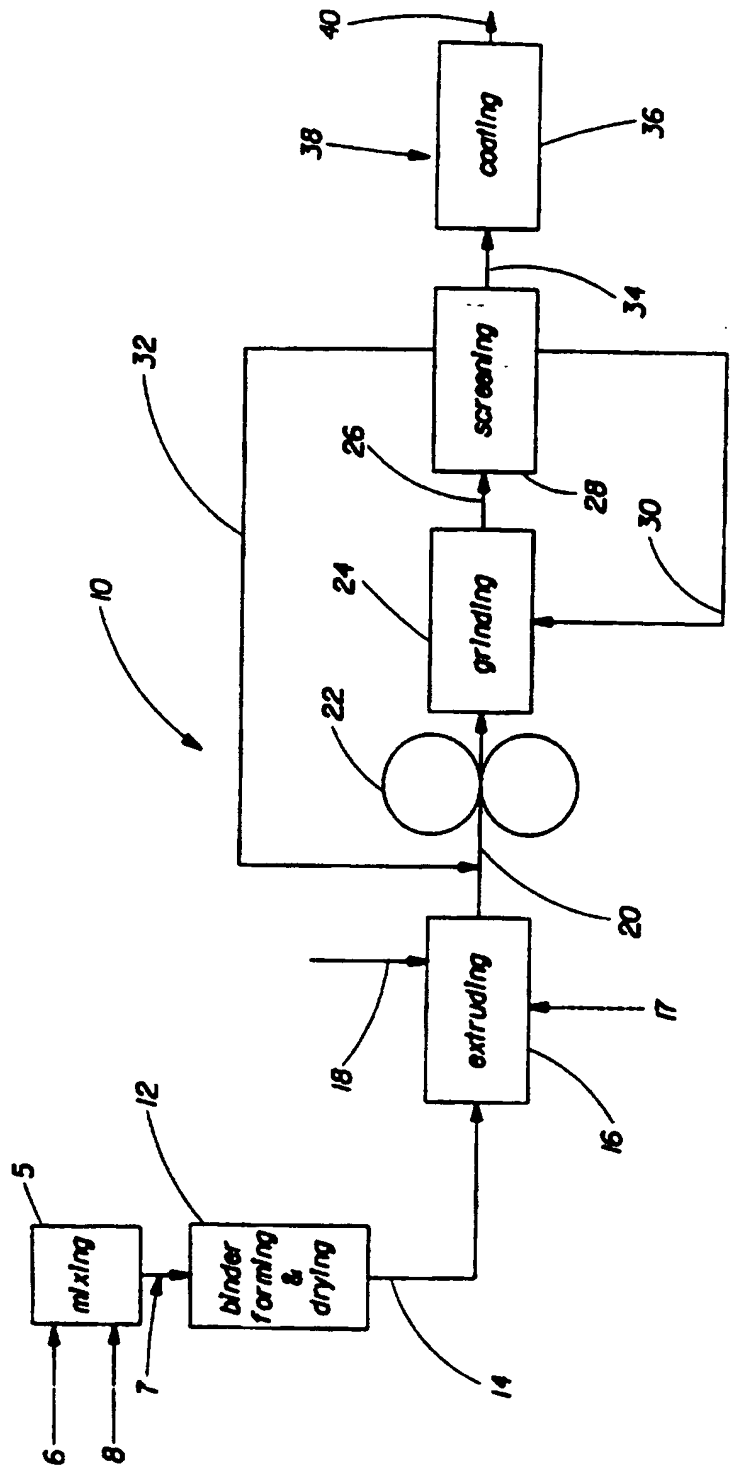
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